United States Department of Agriculture,

BUREAU OF CHEMISTRY,

H. W. WILEY, Chief.

THE TESTING OF CHEMICAL REAGENTS.

A. O. A. C. COOPERATIVE WORK, 1905.

At the regular annual meeting of the Association of Official Agricultural Chemists held in November, 1903, a committee was appointed to cooperate with a similar committee of the American Chemical Society in an investigation of the quality of chemicals used for analytical purposes. The first report was submitted at the annual meeting held in St. Louis, September, 1904, and is given in full in the proceedings of that convention.[1] The committee regretted exceedingly to be compelled to report that only two chemists, aside from those in the Bureau of Chemistry, took any active part in the work and sent in reports. The A. O. A. C. committee desires the assistance of as many chemists as possible, whether members of this Association or not, in this work. The committee invites suggestions as to what chemicals should be tested, the degree of purity desired, and the nomenclature to be employed. Shall we retain the designations "C. P.," "pure," and "commercial," or shall a new classification be made, and in either case what shall be the basis of standards?

Below will be found a list of twenty chemical reagents to be tested as received in original packages from the dealers. The report should include the names of the manufacturers. The observations to be made are specified, and the following books are to be used unless otherwise indicated: "Die Prüfung der Chemischen Reagentien," third edition, by C. Krauch, or a translation of the same; "The Testing of Chemical Reagents," by J. A. Williams and L. W. Dupré, published by D. Van Nostrand Company, 23 Murray street, New York, N. Y., and the 1900 United States Pharmacopœia (U. S. P.). In case two different tests are given for the same impurity, the one to be followed is specified. All calculations are to be based on the international atomic weights with hydrogen as the unit.

ACETIC ACID, GLACIAL, 99.5 PER CENT.

Physical appearance; specific gravity[2] at 20° C.; congealing point; per cent of acid by titration, using phenolphthalein and methyl-orange as indicators in sep-

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[1] U. S. Dept. Agr., Bureau of Chemistry, Bul. 90, p. 157.

[2] In the eighth edition of the United States Pharmacopœia, which is in press, 25° C. has been adopted as the temperature for taking specific gravities.

arate solutions; miscibility in water and alcohol; nonvolatile matter in 50 cc on steam bath and also by subsequent ignition; presence of hydrochloric acid and sulphuric acid in a 10 per cent solution after twelve hours standing; presence of salts of aluminum, copper, lead, calcium, and iron in 10 per cent solution. (Saturate a 10 per cent acid solution with ammonium hydroxid, 0.900 sp. gr., no change within twenty-four hours; to separate portions of alkaline solution, add ammonium sulphid solution and ammonium oxalate solution, no change should be manifest.) Presence of acetone, etc., by iodoform test in 10 per cent solution (Richter). Presence of sulphurous and formic acid (U. S. P.); empyreuma, by potassium permanganate solution (Krauch); render alkaline with potassium hydroxid solution and note odor (U. S. P.).

Dissolve 20 mg powdered potassium bichromate in 10 cc of concentrated sulphuric acid, add 10 cc of acetic acid and mix well. No green color should develop within one-half hour.

ALCOHOL, ETHYL, ABOUT 95 PER CENT.

Physical appearance; specific gravity at 20° C.; miscibility in water; nonvolatile matter in 100 cc, on steam bath and by ignition after evaporation; foreign odor like fusel oil (U. S. P.); aldehyde or tannin (U. S. P.); organic impurities by silver nitrate test (U. S. P.); furfurol (Krauch); reaction to litmus paper and the number of cubic centimeters of hundredth-normal potassium hydroxid solution required to neutralize the acidity of 100 cc of the alcohol, using phenolphthalein as indicator. (Before titrating, dilute the alcohol with an equal volume of water, whose acidity to phenolphthalein has been determined. Deduct the acidity of the water from the total acidity of the mixture of alcohol and water, which gives the acidity of the alcohol.)

AMMONIUM HYDROXID.

Physical appearance; specific gravity at 20° C.; per cent of ammonia gas by titration, using cochineal and methyl-orange as indicators in separate portions; miscibility in water; nonvolatile matter in 50 cc, on steam bath and by ignition after evaporation; presence of chlorid and sulphate in 10 per cent solution after twelve hours standing; presence of salts of calcium, zinc, copper, and lead; carbonic acid; empyreumatic bodies; arsenic, Marsh-Berzelius method, running one hour.

ETHYL ETHER.

Physical appearance; specific gravity at 20° C.; miscibility in 95 per cent alcohol and in chloroform; boiling point; foreign odor; nonvolatile matter in 100 cc on steam bath; reaction to litmus; acidity to phenolphthalein in 100 cc. (Determine as directed under alcohol. The water and the ether are not completely miscible, consequently it is best to make the titration in a glass-stoppered flask, shaking vigorously after each addition of hundredth-normal potassium hydroxid solution.) Aldehyde, etc. (U. S. P.). Hydrogen peroxid and ozone (Krauch). (The potassium iodid must be free from iodate, and the liberation of free iodin is to be determined by a few drops of starch solution.) Alcohol and water in excess (U. S. P.). (The latter observation is to be made at about 20° C.) Water (Krauch).

HYDROCHLORIC ACID.

Physical appearance; specific gravity at 20° C.; per cent of acid by titration, using phenolphthalein and methyl-orange as indicators in separate portions; miscibility in water; nonvolatile matter in 50 cc, on steam bath and by ignition after evaporation; sulphuric acid in 10 per cent solution after twelve hours standing; presence of salts of copper, iron, aluminum, calcium, and lead;

presence of chlorin (Krauch), except that instead of using a starch solution, add a few drops of carbon disulphid and shake; bromid and iodid, liberate bromin or iodin with chlorin water, add carbon disulphid and shake; sulphurous acid (Krauch); arsenic by the Marsh-Berzelius method, running one hour.

HYDROGEN DIOXID.

Physical appearance; number of cubic centimeters of tenth-normal potassium hydroxid required to neutralize acidity of 50 cc by direct titration, using phenolphthalein as indicator. Amount of nonvolatile matter in 50 cc, by evaporation on steam bath and complete drying at 120° C. (Note any foreign odor during or after evaporation.) Test for the presence of salts of aluminum, barium, calcium, iron, lead, magnesium, potassium, and sodium as follows: evaporate to dryness 25 cc previously rendered alkaline with ammonia water, take up the residue with 25 cc of water, acidulated with hydrochloric acid, and test separate portions for the different metals. No change of color or precipitate should result either immediately or after standing twelve hours when treated with hydrogen sulphid gas, or ammonia water, except when certain metals and a phosphate are present; or when treated with ammonia water and ammonium sulphid, except when certain metals and a phosphate are present; or with an ammoniacal portion acidulated with acetic acid, then treated with ammonium oxalate, and allowed to stand for twelve hours; or with ammonia water and magnesia mixture; or dilute sulphuric acid and twelve hours standing; or testing with the flame test. Arsenic, render 25 cc alkaline with ammonia water, evaporate to dryness on steam bath, take residue up with dilute sulphuric acid, and treat according to the Marsh-Berzelius method for one hour. Boric acid, Bulletin No. 65, Bureau of Chemistry, page 110, method (a). Hydrofluoric acid (U. S. P.). Phosphoric, sulphuric, and hydrochloric acids by the usual tests, using original solution and allowing to stand for twelve hours. Silicate, acidulate 25 cc with hydrochloric acid, evaporate to dryness on steam bath, dry at 100° C. for one hour; the resulting residue should be completely dissolved in 100 cc of 5 per cent hydrochloric acid. Per cent of absolute hydrogen peroxid (U. S. P.). (Each cubic centimeter of tenth-normal potassium permanganate corresponds to 0.00169 gram of hydrogen peroxid.)

NITRIC ACID.

Physical appearance; specific gravity at 20° C.; per cent of acid by titration, using phenolphthalein as indicator; miscibility in water; nonvolatile matter in 50 cc, on steam bath and by ignition after evaporation; sulphuric and hydrochloric acids in 10 per cent solution after twelve hours standing; presence of copper, lead, calcium, and iron (U. S. P.); hyponitrous acid (Krauch); bromin and bromic acid and iodin and iodic acid (U. S. P.); arsenic by Marsh-Berzelius method, running one hour.

OXALIC ACID.

Physical appearance of chemical and 10 per cent aqueous solution; amount and nature of nonvolatile matter on ignition, in 5 grams; sulphate, chlorid, phosphate, nitrate (by indigo test) in 10 per cent solution and twelve hours standing. (Acidulate strongly with nitric acid when testing for chlorid.) Presence of salts of aluminum, calcium, iron, lead, magnesium, potassium, and sodium by methods given under hydrogen dioxid; ammonia, both with sodium hydroxid and Nessler's reagent (Krauch); color of solution when 1 gram of the acid is mixed with 10 cc of concentrated sulphuric acid and gradually heated to 50° C. Per cent purity by titrating with standard solution of potassium permanganate.

POTASSIUM BICHROMATE.

Physical appearance of chemical and of a 10 per cent aqueous solution; loss of weight on heating powder at 120° C. for six hours; chlorid, phosphate, and sulphate in 5 per cent solution after twelve hours standing. (It is necessary to acidulate strongly when testing for sulphate.) Treat a 5 per cent solution with enough alcohol and hydrochloric acid to completely reduce the chromium to the basic condition, test this solution, after dissipating aldehyde and excess of alcohol, by the regular group reagents for aluminum, iron, magnesium, calcium, sodium, and strontium. Per cent of purity by the iodometric method, which consists in liberating free iodin from an iodid by the bichromate in a solution acidulated with sulphuric acid and titrating. (Classen Analyt. Chemie, Vol. I, page 376.) The decinormal factor of potassium bichromate is 0.00487111.

POTASSIUM CARBONATE.

Physical appearance of chemical and of a 10 per cent aqueous solution; moisture at 130° C. for six hours; foreign metals, by outline given under hydrogen dioxid, aluminum, calcium, iron, lead, zinc, magnesium, and sodium; arsenic by Marsh-Berzelius method; chlorid, phosphate, sulphate, silicate, sulphid (Krauch); cyanid and thiosulphate (U. S. P. 1890). Per cent of purity based on anhydrous product, using methyl-orange as indicator.

POTASSIUM HYDROXID BY ALCOHOL.

Physical appearance; solubility in water and in 95 per cent alcohol; note initial color and change of color on standing of 10 per cent alcoholic solution. (First ascertain whether there be any disturbing agent present in the alcohol.) Per cent of alkali on titration, using phenolphthalein as indicator; presence of salts of calcium, iron, aluminum, lead; chlorid, sulphate, nitrate, carbonate, silicate (Krauch); presence of sodium compound in potassium hydroxid by flame test and U. S. P. 1890; ammonium compound in 10 per cent solution, by Nessler's reagent; arsenic in 5 grams, by Marsh-Berzelius method, one hour running. Test for the presence of phosphate by means of ammonium molybdate solution.

POTASSIUM HYDROXID (COMMONLY KNOWN AS PURE).

Test according to U. S. P. requirements but test also for arsenic by Marsh-Berzelius method and for the presence of phosphate by means of ammonium molybdate solution.

POTASSIUM IODID.

Physical appearance of chemical and of a 10 per cent aqueous solution; number of cubic centimeters of tenth-normal sulphuric acid required to neutralize alkalinity of 5 grams; foreign metals in 10 per cent solution acidulated with hydrochloric acid, as outlined under hydrogen dioxid, barium, calcium, iron, lead, sodium, and magnesium; arsenic by Marsh-Berzelius method; sulphate (Krauch), cyanid, nitrite and nitrate (use 5 grams and other ingredients in proportion), bromid, chlorid, and thiosulphate (U. S. P.). Iodate, acidulate a 10 per cent aqueous solution (prepared with freshly boiled and cooled water) with sulphuric acid free from nitrons and sulphurous acid, add carbon bisulphid, and shake. (A simple darkening does not indicate iodate.) Per cent purity (U. S. P.).

POTASSIUM PERMANGANATE.

Physical appearance; solubility in water; presence of nitrate (U. S. P.); chlorid and sulphate (Krauch); sodium salt by flame test. Arsenic by Marsh-Berzelius method. (Decolorize aqueous solution containing 5 grams with alcohol, filter, evaporate to dryness on steam bath, take up residue with dilute sulphuric acid, and transfer to generator.) Per cent of purity with oxalic acid.

SILVER NITRATE.

Physical appearance of chemical and of a 10 per cent aqueous solution; reaction to litmus paper; presence of copper, lead, and other foreign salts (U. S. P.). Per cent of purity determined by gravimetric chlorid method.

SODIUM CARBONATE, ANHYDROUS.

Physical appearance of salt and of a 10 per cent aqueous solution; per cent of moisture, drying at 130° C. for six hours; presence of salts of ammonium, aluminum, arsenic, calcium (ammonium oxalate test), lead, and potassium (Krauch); chlorid, phosphate, silicate, sulphate, thiosulphate, and free alkali (Krauch). Per cent of purity based on product as received, using methyl-orange as indicator.

SODIUM HYDROXID, BY ALCOHOL.

Physical appearance; solubility in water and in 95 per cent alcohol; note initial color and change of color on standing of 10 per cent alcoholic solution. (Ascertain purity of alcohol first.) Per cent of alkali on titration, using phenolphthalein as indicator; presence of salts of calcium, iron, aluminum, and lead; chlorid, nitrate, carbonate, and silicate (Krauch); ammonium compounds in 10 per cent solution of alkali, by Nessler's reagent; arsenic in 5 grams, by Marsh-Berzelius method, one hour running. Test for the presence of phosphate by means of ammonium molybdate solution.

SODIUM HYDROXID (COMMONLY KNOWN AS PURE.)

Test according to U. S. P. requirements but add test for arsenic by Marsh-Berzelius method and for the presence of phosphate by means of ammonium molybdate solution.

SODIUM THIOSULPHATE.

Physical appearance of chemical and of a 10 per cent aqueous solution; reaction to phenolphthalein; presence of foreign metals in 10 per cent solution; calcium (Krauch); lead, etc. (by hydrogen sulphid); aluminum, iron, etc. (ammonia water and ammonium sulphid). Sulphate (Krauch), iodin solution must be free from sulphate.

SULPHURIC ACID.

Physical appearance; specific gravity at 20° C.; per cent of acid by titration, with phenolphthalein as indicator; nonvolatile matter in 50 cc after evaporation and ignition; miscibility in 4 to 5 volumes of water and alcohol (95 per cent); nitrous acid and nitric acid (U. S. P.); sulphurous acid (U. S. P.); presence of copper, lead, iron (U. S. P.); selenium (Krauch); halogens and ammonium compounds (Krauch); arsenic by Marsh-Berzelius method, running one hour.

Those who will take part in the work are requested to inform the chairman of the committee to that effect as soon as possible, and to report results to him not later than October 1, 1905.

L. F. KEBLER, *Chairman.*
A. L. WINTON,
B. W. KILGORE,
A. O. A. C. Committee on the Testing of Chemical Reagents.

WASHINGTON, D. C., *May 27, 1905.*

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